B-Tech Thesis 2015

FABRICATION OF FUNCTIONALLY GRADED MATERIAL (FGM) SET-UP

A thesis submitted in fulfillment of

the requirements for the degree of

BACHELOR OF TECHNOLOGY

IN

MECHANICAL ENGINEERING

BY

DEEPAK KUMAR SAHOO

ROLL-111me0341

Department of Mechanical Engineering, NIT, Rourkela

Department of Mechanical Engineering

National Institute of Technology

Rourkela-769008

National Institute of Technology NIT Rourkela

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DEEPAK KUMAR SAHOO (111ME0341)

Under the supervision of

Prof. S.C.MOHANTY

Associate Professor

Department of Mechanical Engineering, NIT, Rourkela

Department of Mechanical Engineering

National Institute of Technology

Rourkela-769008

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CERTIFICATE

This is to certify that the work in this thesis entitled: "DESIGN AND FABRICATION OF AN EXPERIMENTAL SET UP FOR FUNCTIONALLY GRADED MATERIALS" by **Deepak Kumar Sahoo** has been strictly carried out under my supervision in partial fulfilment of the requirements for the degree of **Bachelor of Technology** in *Mechanical Engineering* during session 2011- 2015 in the Department of Mechanical Engineering, National Institute of Technology, Rourkela.

To the best of my knowledge, this work has not been submitted to any other University/Institute for the award of any degree or diploma.

Dr. S.C.Mohanty

(Supervisor)

Associate Professor

Dept. of Mechanical Engineering

National Institute of Technology

Rourkela - 769008

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DATE: 10-05-2015

PLACE-ROURKELA

DEEPAK KUMAR SAHOO

Roll No. – 111ME0341 8th Semester, B. Tech Mechanical Engineering Department National Institute of Technology Rourkela-769008

CONTENTS

Index of Figures

List of Tables

ABSTRACT

Functionally graded materials (FGMs) are advanced composites with graded properties that are novel concepts in the structural design of materials and have extensive applications in the aerospace engineering, aircrafts and automotive industry. FGMs with the continuous variation of microstructures and properties are expected to have the reduction of residual and thermal stresses. In case of metal-ceramic FGMs advancement over heat and corrosion resistance of ceramics and mechanical strength of metals in such applications as thermal or chemical barriers. FGMs can also be designed to take.an improved adhesive bonding strength between metals and ceramics. Multifunctional metal-ceramic structures are suitable for applications where toughness and hardness are two essential requirements for engineering and scientific research applications. The objective of the research paper is to fabricate graded metal-ceramic composites in bulk manufacturing processes for commercial applications. This paper aims at control of the evolution of mechanical properties through the use of a nanoparticle sintering aid and fabrication of graded metal-ceramic composites using pressureless sintering and bulk molding technology. A new sintering aid TiO_2 was introduced to control the evolution of mechanical properties of graded Nickel-Alumina composites. The evolution of mechanical properties was then used in a recently developed two-dimensional micro thermo-mechanical analysis to determine the effects of develop gradient architectures. Finally, a laboratory-scale FGM setup is designed based on a powder metallurgy (PM) and Rapid Prototyping (RP) technology to process geometrically complex gradient structures.

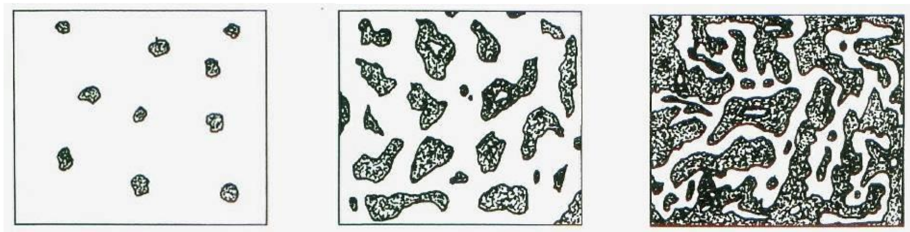
Keywords: Alumina, Nickel, TiO_2, Rapid Prototyping (RP), Powder Metallurgy (PM), Quick Return Mechanism (QRM)

CHAPTER 1

Introduction

1.1 Background and Motivation

Multifunctional and multi-material structures combine material properties to meet the required functional requirements. These materials are layered one by one, but each layer having different properties and then bonded with strong adhesive. However, this sharp division of materials at the bonding zone generates a weak interface that results in stress concentration. For example, an impact-generated stress wave that propagates through the sharp interface creates a tensile force as the wave transmits across and reflects off the interface [1]. The resultant stress generated can lead to the deboning of the joined materials at the weak interface. The solution to this problem is to transition the interface through a material gradient. Functionally graded materials (FGMs) spatially vary composite microstructure or composition to optimize the resultant material properties. The microstructural distribution of phases within a two-phase material can be identified by three basic types of morphologies: (1) dispersed, (2) aggregated, and (3) percolated [2]. As shown in Figure 1.1a, a dispersed grain structure occurs when the volume fraction of one phase is low, then discretely and randomly distributed within the dominant matrix phase. As the volume fraction of the inclusion phase more, particle agglomerates begin to form an aggregated structure as shown in Figure 1.1b. With even further increasing volume fraction, the phase will stand at a critical point called the percolation threshold. At this point, a long-range interconnectivity begins to occur between particle agglomerates as shown in Figure 1.1c.



(a) (b) (c)

Figure 1.1: Basic types of heterogeneous two-phase microstructures: (a) dispersed grain structure, (b) aggregated grain structure, and (c) percolated cluster structure [2].

Compositional gradients take the advantage of multiple material properties and remove the stress concentrations that occur at sharp interfaces of different materials. The microstructural properties vary with the position within the gradient and can be used to tailor composite performance and functionality. For instance, a functionally graded plate may be used in applications of high temperature and stress whereas a thermal resistant material on the exposed outer surface is graded with an inner material that exhibits better mechanical properties. A two-phase graded structure, consisting of two pure base materials at the ends and a gradient microstructure in between them is shown in Figure 1.2. The gradient can be depicted through homogenization as a linearly varying composition, V(x), from x equal 0 to L.

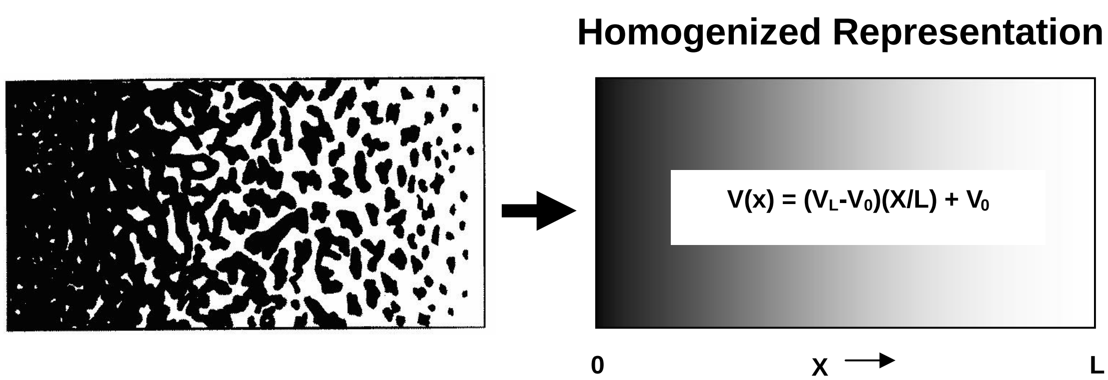


Figure 1.2: Two-component graded structure, for which the microstructure varies linearly [2].

Natural structures like bamboo and bone, also exhibit graded material distributions that optimize performance through spatial variations. Bamboo utilizes a hierarchical microscopic gradient structure of bundle sheath fiber reinforcement to provide radial strength to bending loads [3]. Another example is bone, primarily made of collagen and calcium phosphate, which has higher strength and hardness. A gradient of pores within the bone is naturally tailored to increase the ductility and permission to fluid passage. In general there are four types of gradation: volume fraction, shape, orientation and size of material as shown in figure 1.3. The structural gradation of the material could be described as a transition function that shows the relation between spatial position and gradient status.

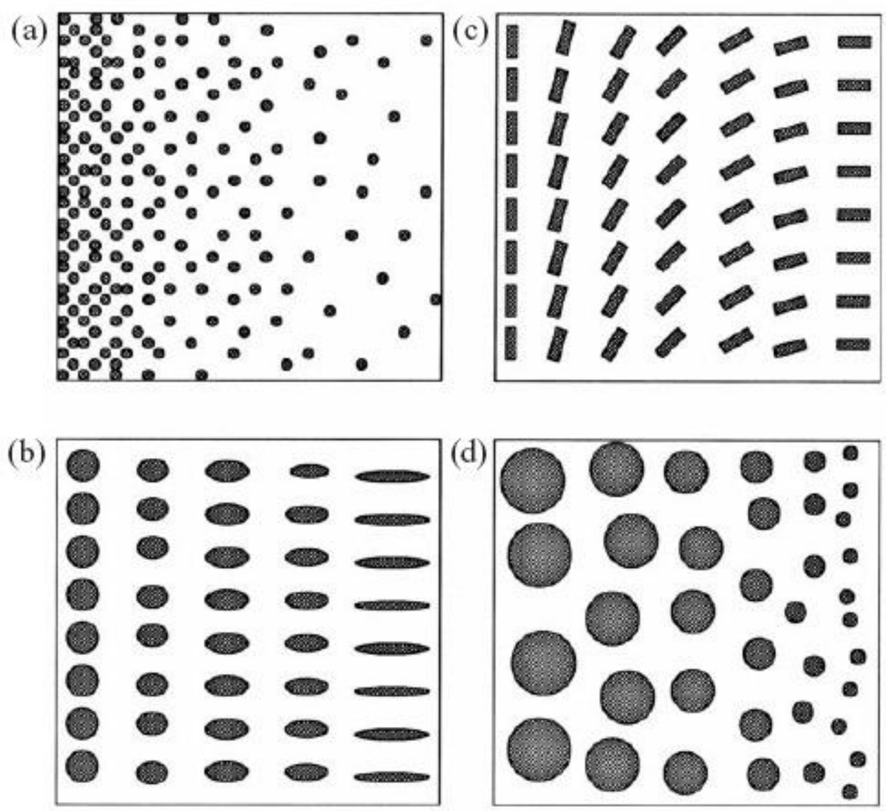


Figure 1.3: Different types of Gradation of (a) volume fraction (b) shape (c) orientation (d) size (Neubrand 2001)

The fabrication of functionally graded materials is obstructed due to the variation of mechanical thermal, chemical, and kinetic properties within the composite. Residual stresses are generated across a material interface due to discontinuities occurs in material structures and properties [4]. Despite these challenges, compositional gradient structures offer significant benefits towards modern industry, research, and scientific areas. In addition to utilizing the advantageous properties of the pure base materials, the transition across materials with thermal expansion mismatch can be smoothed [5, 6].

This section will introduce the challenges and methods of fabricating experimental set up for functionally graded metal-ceramic structures. This section will also discuss novel techniques for reducing fabrication costs and increasing fabrication production through bulk processing techniques using powder metallurgy and rapid prototyping.

1.2 History of Functionally Graded Materials

The idea of compositional and structural gradient in material microstructure was first suggested for composites and polymeric materials in 1972. Bever studied various gradient composites by investigating the global material structures and properties and reviewed potential applications of graded composites. Shen (1972) reported gradation of polymeric material might be induced by the variation of chemical nature of the monomers, the constitution of the polymers at molecular level and the supramolecular structure. The effective properties, such as chemical, mechanical, thermal, biomedical, transport properties and applications in gasoline tank and damping materials were considered. However, the design, experimental setup fabrication and evaluation of this gradient structure was not studied. Until 1985, the use of continuous texture control was presented to improve the adhesion strength and reduce the thermal stress on ceramic coatings and joints being developed for a reusable rocket engine. The term functionally graded materials was coined for these gradient composites and materials for more accurate description. The capabilities of FGMs are achieved to withstand a high surface temperature of 1700^{0}C and a temperature gradient of 1000^{0}C across only a 10mm section. The development of this research project was spread worldwide via media, papers and international conferences. Potentially applicable fields for FGMs are shown in figure 1.4.

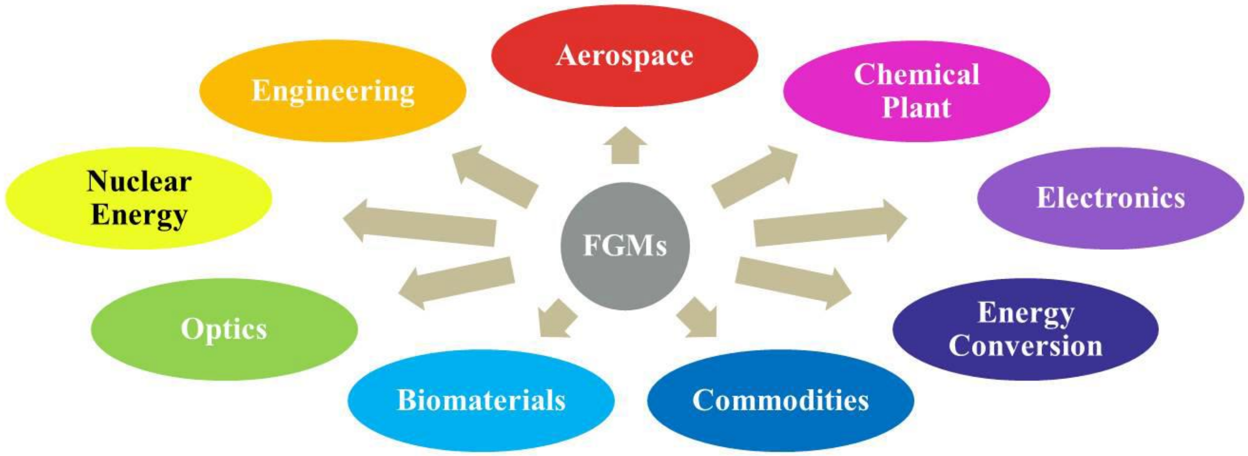


Figure 1.**4:** Potentially applicable fields for FGMs

1.3 General views about FGMs

The properties of metals or ceramics strongly depend on the nature of adhesive bonding such as bonding strength. Ceramics typically exhibit high hardness, low density, and brittleness, withstand high temperature, creep, resistivity towards corrosion, radiation, wear and shock resistance whereas metals are typically ductile in nature, high tensile strength, high toughness, and more density. Multi-layer and Multifunctional metal-ceramic structures are well suited for applications where both toughness and hardness are the primary entities. For example, knives and cutting tools must be hard on the cutting edge. The introduction of a material gradient structure to replace a sharp interface presents new fabrication challenges for experimental setup. The graded interface varied in a continuous manner in composite microstructure from one material phase to another. In order to simplify the fabrication techniques, this continuous transition in microstructure is often approximated by discrete layers of distinct composite compositions, as seen in Figure 1.6. Properties of these composite compositions are often developed and then modeled by rule-of-mixtures (ROM) or modified ROM formulations [2, 6, 12]. However, the introduction of the material gradient to replace the sharp interface does not eliminate the formation of stresses due to the different material properties of both the materials.

In fact, for certain geometries, the use of an arbitrary gradient may result in higher local stress concentrations [13]. Consequently, gradients must be carefully tailored to reduce the formation of these stresses, as presented in the following section.

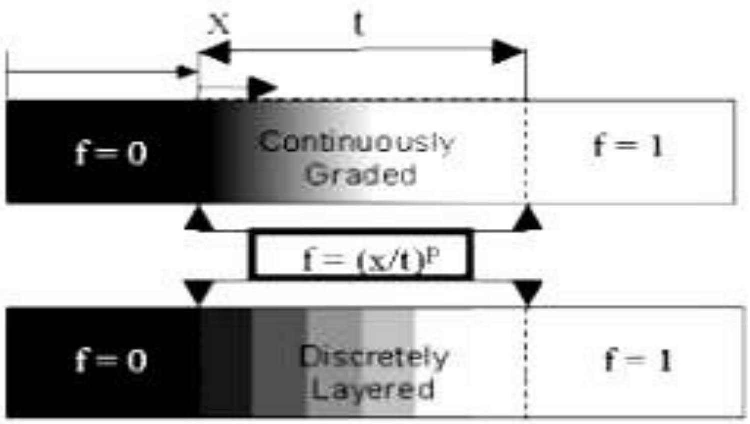


Figure 1.5 graded structure

1.4 Different techniques used for fabrication of FGMs

During the FGM program in Japan from 1987 to 1991, several processing methods were developed for FGMs parts as a thermal barrier of a space plane. These methods included powder metallurgy, plasma spraying, physical and chemical vapor deposition, self-propagating high temperature synthesis (SHS) and galvanic forming. Since 1991, various new methods have been developed by novel thinking of researchers. The processing of FGMs has been classified in several ways in review papers. As shown in figure 1.6 Miyamoto et.al. (1999) classified the fabrication of FGMs into four categories including bulk, layer, perform and melt processing.

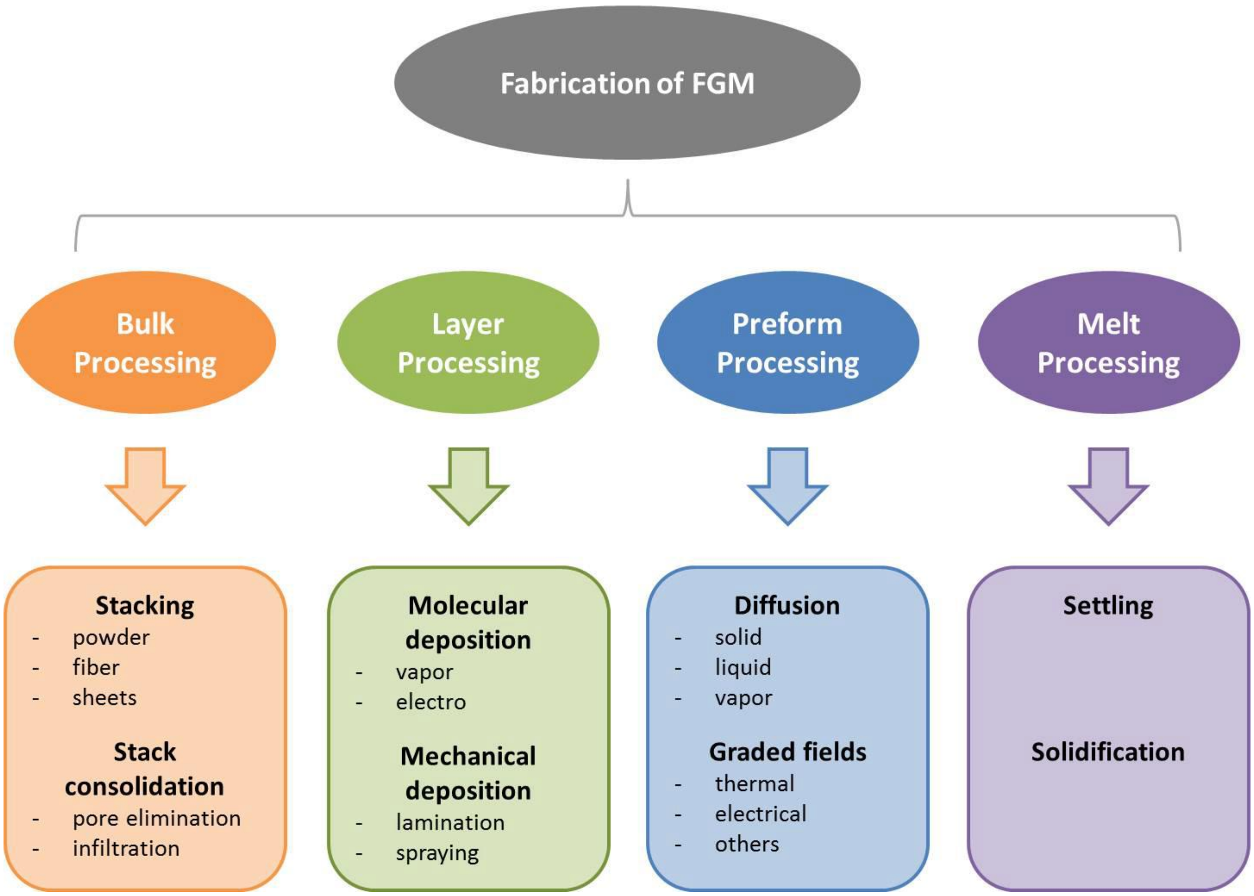


Figure 1.6: Proceeding methods and classification for fabrication of FGMs

1.5 Scope of Research

This thesis investigates the fabrication of graded metal-ceramic composites using pressureless sintering with a new nanoparticle sintering aid and focuses on the procedures and results of using a nanopowder TiO_2 as sintering aid for Nickel-Alumina composites. The use of sintering aid for graded composite structures had not previously been investigated. So, it was necessary to quantify the nanoparticle effects on the evolution of mechanical properties in gradient architectures. No prior research effort has been conducted for the evolution of porosity reduction when nanoparticle sintering aids are employed.

The focus of the current research work depicts the development of commercial powder processing and rapid prototyping techniques to fabricate geometrically-complex graded plate specimens. In this thesis, the design and fabrication of a laboratory-scale processing system based on commercial concrete molding technology is presented. An initial investigation into the processing of graded metal-ceramic composites was initiated using a variety of layered structures that were qualitatively analyzed for cracking.

1.6 Research Objective

The objective of this current research is to advance the understanding of graded metal-ceramic composite fabrication to enable the commercial processing of products from graded metal-ceramic composites. Despite this challenge, conventional powder processing techniques and pressureless sintering have been conducted using Nanopowder TiO_2 as a new sintering aid to control the evolution of shrinkage strains and mechanical properties of gradient structures. A systematic effort was undertaken to produce FGMs specimens from Nickel and Alumina powders. These composite samples were produced over a wide range of phase volume fractions in both homogeneous forms to quantify sintering behavior and to investigate cracking from shrinkage-induced stresses during the sintering process. The basis for the processing of graded metal-ceramic products, a commercial technology had to be developed and fabricated to study the effects of processing conditions.

First, this research will examine the effects of a Nanopowder sintering additive applied to functionally graded specimens. After compaction sintering behavior, microstructural changes and characteristic properties will be documented. Secondly, commercial processing methods will be investigated to produce geometrically complex graded metal-ceramic specimens, and the goal of this research is to develop a laboratory-scale molding assembly similar technology developed in case of concrete industry. The primary focus is to advance the metal-ceramic FGM production by developing novel technologies and following the procedures for reduction of fabrication complexity and cost. For this reason, conventional powder processing techniques and pressureless sintering will be carried out.

CHAPTER-2

2 Literature Review

Several methods have been adopted to design and fabricate complex graded materials called functionally graded materials. Pines M [1] reported about Pressureless Sintering process of Powder to design Functionally Graded Metal-Ceramic Plates. Markworth A.J. et.al. [2] reviewed on modeling application to design functionally graded materials. Amada S. et.al. [3] studied about mechanical structures of bamboos in view front of functionally graded materials. Rabin B.H. et.al. [4] analyzed the residual stresses developed between dissimilar metals. Mortensen A. and Suresh S. [5] reported on the processing techniques of functionally graded metals and metal-ceramic composites. Chin E.S.C. [7] developed a functionally graded armor composites for army focused research team. Rabin B.H. and Williamson R.L. [8] presented the skills for Design and Fabrication of Metal ceramic Gradient Materials. Watanabe R. [9] studied on power metallurgy techniques to develop functionally graded materials. Drake J.T. et.al. [10] worked on finite element model to analyze thermal residual stress at the interface of metal-ceramic graded materials and then optimize for residual stress reduction. German R.M. [11] discussed on sintering theory and sintering behavior of functionally graded materials. Han C.Z. et.al. [12] studied on Effect of Powder Characteristics on Microstructural Development in products Produced by less Sintering process of Al/TiO2 Composite Powder. Winter A.N. [13] reported on Fabrication techniques of Graded Nickel-Alumina Composites with a Thermal-Behavior-Matching Process. Olhero S.M. and Ferreira J.M.F. [14] analyzed the effect of Different Oxide Additives on Colloidal Processing and Sintering of Alumina. Erkalfa H. et.al. [15] discussed the effect of sintering aids MnO2 and TiO2 Additives on Densification process of Alumina at 1250^0C. Zang L.K. et.al. [16] experimented on pressureless sintering process Nickel-Alumina composite ceramics. Acchar W. and Fonseca J.L. [17] studied to know sintering behavior of alumina reinforced with (Ti, W) carbides. Rabin B.H. and Heaps R.J. [18] worked on Powder Processing of Ni-Al2O3 FGM and focused on their characteristics. Pines M.L. and Bruck H.A. [19] reported on Pressureless sintering process of particle reinforced metal-ceramic composites for functionally graded materials and aimed at

Porosity reduction models. Shabana Y.M. et. al. [21] studied on modeling the evolution of stress due to differential shrinkage in case of powder-processed functionally graded metal-ceramic composites during the pressureless sintering process. Kumar P. et.al. [22] investigated on the deposition of fine powder particles through hopper-nozzles applied to multilateral solid freeform fabrication. Shelare S.D. and Handa C.C. [23] studied on Performance Based Tolerance Scheme for quick return motion Mechanism. Thakare P.S., et.al. [24] worked on computer aided modeling and position analysis of crank slotted bar mechanism. Ding H. et.al. [25] presented computer-aided structure decomposition theory of kinematic chains and its applications to modeling of different mechanisms.

CHAPTER 3

Fabrication of Functionally Graded Metal-Ceramics

This section will discuss popular fabrication techniques of functionally graded metal-ceramic structures. The design of compositional gradients has been manifested by different fabrication methods. In general, this research work will focus on powder metallurgy processing techniques and the appropriate additive manufacturing process.

A number of models are available for the design of the spatial variation of the gradient compositions [2]. One of the modern gradient function is the power model law distribution used by Drake *et al.* [10]. Using the power model law distribution, gradients can be customized to be more sensitive base material using either a concave upward or concave downward function.

3.1 Powder Processing

The fluid-like the behavior of powdered materials allows for a variety of green shaping processes, such as die compaction. In addition to that there is a great deal of microstructural and compositional control when working with powder particles. Conventional powder metallurgy processing technique produces a green body of powder with the desired gradient of phase volume fractions. This research mainly focuses on CATIA design and fabrication processes. These processes vary from simple step-wise layering methods that can control mechanically to highly automate continuous distribution operations controlled by using some electronics circuits. The discrete layering process forms the green body by sequentially adding pre-mixed powder compositions to a compaction die. Alternatively, continuous distribution methods attempt to abolish compositional interfaces altogether by gradually increasing phase content in some functional distribution. This object is typically achieved by a highly controlled spray techniques. Layering techniques offer the ability to build up the graded region incrementally without the need for enlightened equipment to continuously monitor the microstructure, which is a critical requirement in continuous grading operations. The green body must be subjected to a solid-state densification process. Densification of FGMs has been achieved by cold isostatic pressing (CIP) and pressureless sintering and by hot isostatic Pressing (HIP) in a closed compact die. Based on previous research [9], for functionally graded structures a powder processing fabrication flow chart is shown in Figure 3.1.

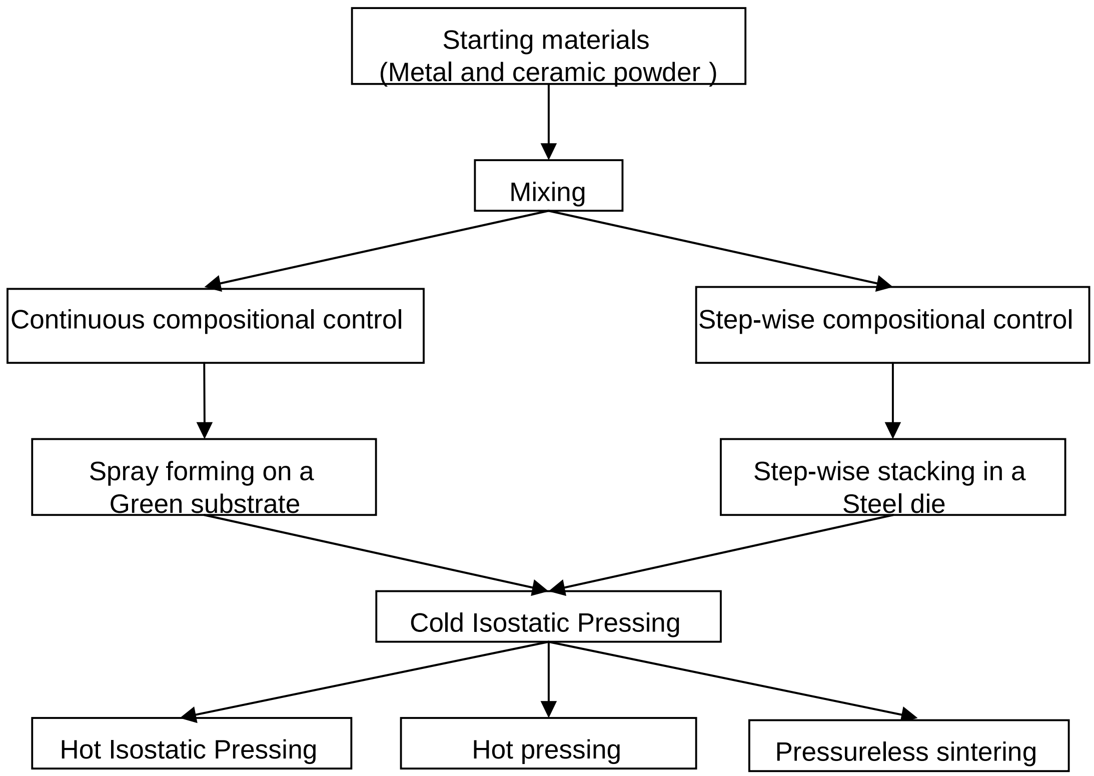


Figure 3.1: Flow chart for the fabrication of functionally graded materials using powder processing.

Powder processing begins with pure Nickel and Alumina powders and finishes as a composite green compact under cold isostatic pressing by the use of steel compaction dies. The creation of a discrete layered graded structure first requires the appropriate mixing of the base materials to powder compositions. Basically, the FGM structure consists of the two base materials on either end of the specimen, with the required compositional gradient layered between the two pure end layers, as shown in Figure 3.2.

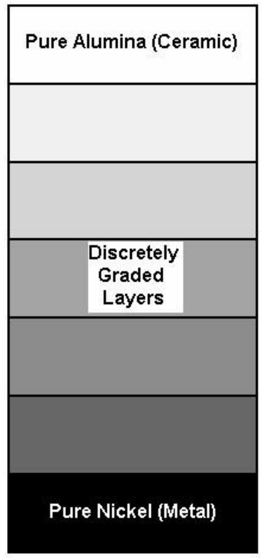


Figure 3.2: Discrete layered FGM structure.

3.2 Powder preparation techniques

Proper mixing time, methods, and available equipment have been varied in an attempt to improve powder mixing process. Previously, success has been achieved by mixing appropriate quantities of Nickel, Alumina and TiO_2 powders for approximately two hours. Powders are properly mixed to form distinct composite compositions that is useful as a homogenous sample. Figure 3.3 documents the flow chart of the entire discrete layering fabrication process.

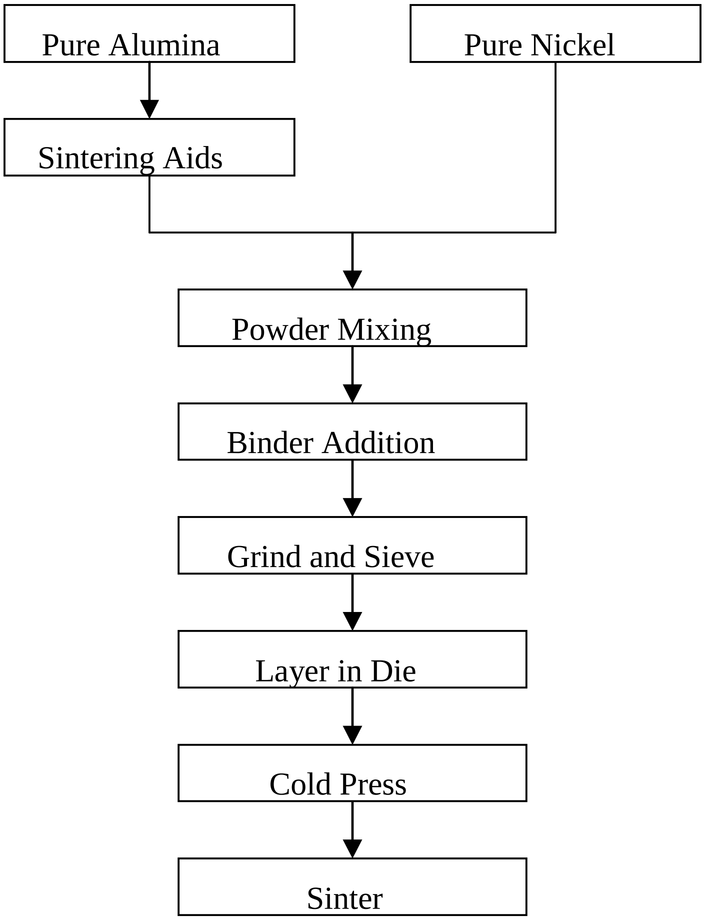


Figure 3.3: Die-based discrete layered powder processing fabrication technique.

3.3 Pressureless Sintering

Sintering is defined as a thermal heat treatment for bonding particles into a coherent (phase difference is zero), mainly solid structure through mass transport events on the atomic scale range [11]. The absence of external pressure applied to the process is known as pressureless sintering. Pressureless sintering is an inexpensive consolidation technique that can produce various sintered geometries. However, in absence of external pressure there is no obstacle for the deformation method of the specimen during sintering, which is the cause of severe differences in differential shrinkage. Despite this challenge, pressureless sintering in a high-temperature furnace provides a simple consolidation method well suited for the manufacturing of graded products multi-materials.

Sinter bonding is highly temperature sensitive. The high sintering temperature is the cause of the acceleration of sintering kinetics. Particle cohesion will be more as the porous interfaces between particles are consumed at increasing temperature. As sintering proceeds, grain growth starts to occur until some maximum is attained, and the sintering process is complete. Particle size also has a significant effect in sintering kinetics. Previous work has been reported that high-energy milling of Aluminum-TiO_2 powder to produce Ti_2O (Al)/Al_2O_3 composites reduces size of particles, which in turn accelerates the densification process [12].

The dominant matrix phase typically controls densification process at low volume fractions of the integration phase. In particular, densification process for metals much faster than ceramics. Consequently, the densification process of FGMs typically exhibits uneven shrinkage between composition layers.

Sintering kinetics can be amended to match the sintering rate of the two powder phases. Particle size control can be used to control sintering rate by modifying specific surface energy. [9, 13]. Alternatively, small quantities of sintering aids are well suited to the ceramic phase to increase the densification rate and to lower the sintering temperature [14-16]. By revising the sintering kinetics and matching the sintering rates of the two phases, FGM warping and cracking while fabrication can be significantly eliminated.

3.3.1 Sintering Schedule

Previous research work has developed a successful pressureless sintering cycle for Nickel-Alumina composites [1, 20]. The sintering temperature is first raised to 150˚C and kept for 30 minutes to let any accumulated moisture to burn off completely. The cycle then raises to 400˚C and is kept for two more hours to burn out the organic binder completely. The cycle ramps to the full sintering temperature of 1350˚C and is kept for four hours. Lastly, the cycle cools down to 0˚C at a particular rate of 100˚C/hr. The entire sintering process was performed under flowing Argon gas to prevent Nickel oxidation. Figure 3.4 displays the full furnace cycle.

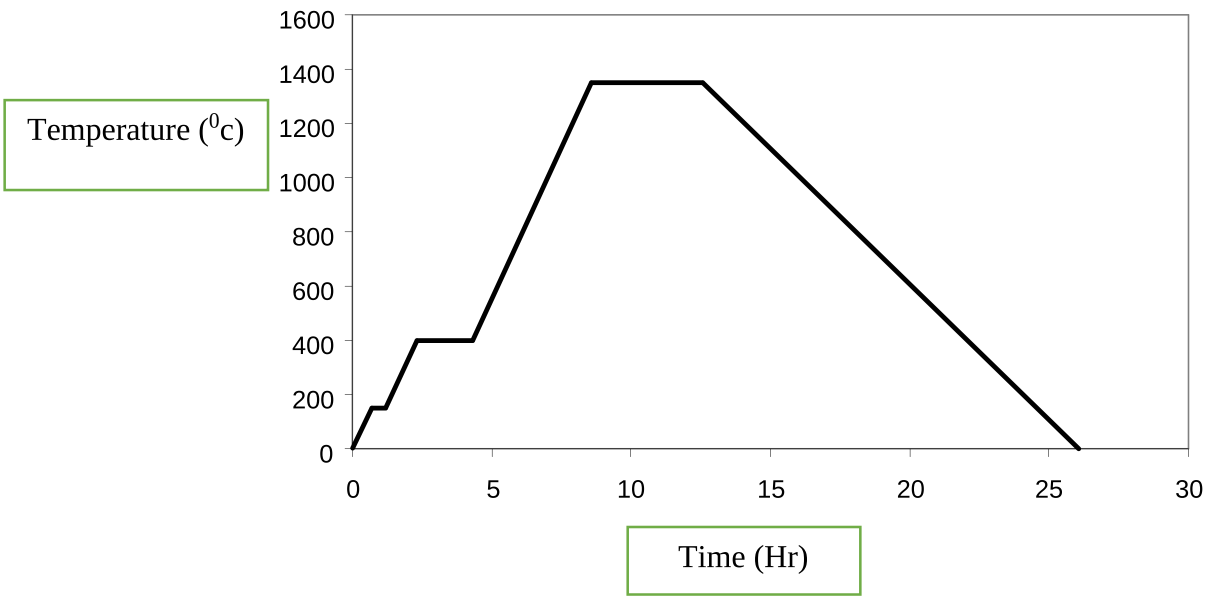


Figure 3.4: Furnace schedule used to pressureless sinter green compacts to final form.

3.3.2 Sintering aid

The sintering of Alumina is an important material in the area of oxide ceramics, usually takes place at high temperatures in the range (1550-1600) ˚C. However, the sintering of metal-ceramic composites is typically limited due to low the melting point of the metal. In the case of Nickel-Alumina composite structures, there is a limitation of sintering temperature is in the range 1350-1400˚C. The ceramics industry has used small amounts of sintering additives, such as TiO_2, MgO, SiO_2, and Fe2O3 to influence the ceramic densification process. [9]. In particular, the hardness of Alumina composites depends on the microstructural grain size and the final density of the sintered samples [17]. Sintering aids are added to ceramics using a doping procedure. Doping processes typically require mixing with dissolved chlorides followed by hydrolyzation with ammonium hydroxide and these are often complicated and time-consuming operations [18]. In this research, the blending of sintering aids through the use of powder mixing will be explored.

Previous experimental observation depicts TiO_2 more efficient than MnO_2 as a sintering aid at lower sintering temperatures with alumina [15]. In this same analysis, Alumina was sintered to 95% theoretical density at 1350˚C using 3 wt. % TiO_2 whereas the pure Alumina sample without sintering additive only reached 77.1% theoretical density at the same temperature. In another experiment, TiO_2 was added to a 10 wt. % Nickel-Alumina powder composite to know the behavior [16]. The Nickel-Alumina composite reached 97% theoretical density by use of 5 wt. % TiO_2 after pressureless sintering at 1480˚C. Conversely, the sintered sample without the TiO_2 additive only reached 60% theoretical density.

3.3.3 Sintering Behavior

Constitutive models often consider metal-ceramic composite powders as a three-phase material consisting of voids, ductile metal particles, and brittle ceramic particles [21]. The behavior of each phase is distinct, in which the metal particles deform elastoplastically, ceramic particles deform elastically, and porosity that is represented by void volume fraction, is directly related to the shrinkage. The sintering process is shown in Figure 3.5 where diffusion between particles forms solid bonds that reduce surface energy by reducing surface area. There is a reduction of porosity as matrix particles sinter between themselves and around the reinforcement particles. Specifically, porosity reductions are observed as sintering begins due to the nucleation of loosely packed particles [21].

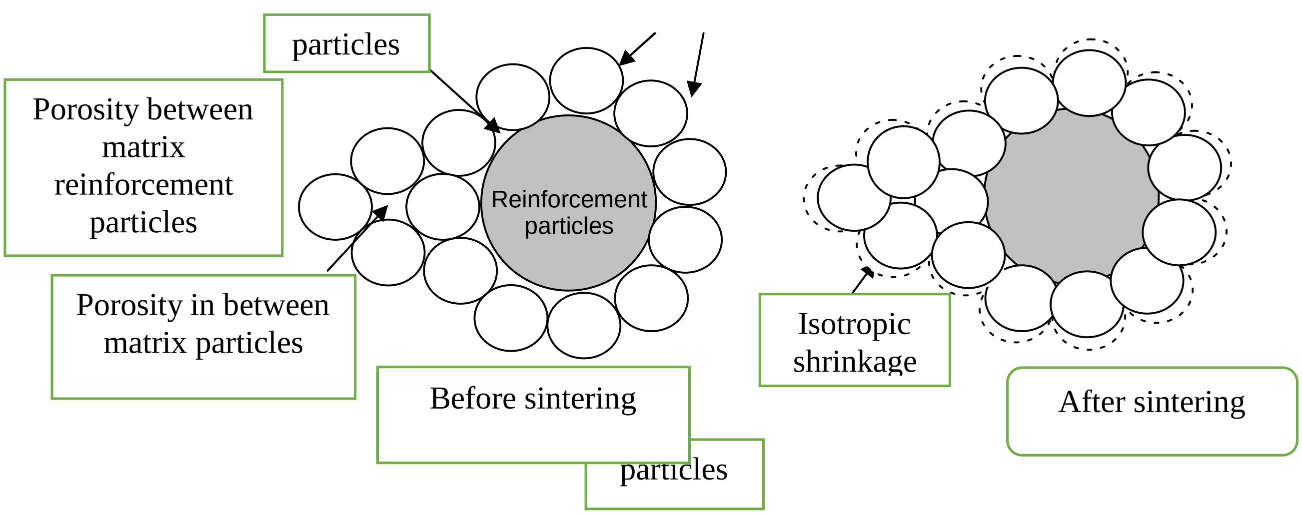


Figure 3.5: Densification behavior of matrix particles, reinforcement particles, and porosity during pressureless sintering [21].

3.4 Molding Specimen fabrication

Traditional Popular bulk molding techniques utilized by commercial powder metallurgy and concrete industries can be chosen to the fabrication of powder processed gradient structures. Combined with the processing techniques reported in the preceding sections, bulk operations can be accomplished by featuring large-scale powder mills and furnaces. Using step-wise discrete layering methods and pressureless sintering, large numbers of graded products could ultimately be produced cheaply within a short period.

3.5 *Scientific and Technical Contributions*

The ceramics engineering sector has long established that certain sintering additives like TiO_2 can be used to lower sintering temperature. This present research applies addition of nanopowder TiO_2 to functionally graded metal-ceramic structures using conventional powder for the first time to reduce sintering temperature. The evolution of shrinkage strains and mechanical properties due to the addition of a nanoparticle sintering aid in metal-ceramic composites has been quantified. Additionally, previously existed models on porosity reduction have been investigated for the prediction of effects of the nanoparticle sintering aid.

The fabrication of crack-free graded cylinders that illustrate higher hardness and less porosity have also been obtained by using nanoparticle sintering aid. Increasing the hardness of the FGMs structure is extremely beneficial to many applications. For instance, in most armor packages the hardest outer material possible will provide the best level of ballistic protection [8]. This research also investigates the processing techniques of geometrically complex graded metal-ceramic plates based on commercial molding technology used in the concrete industry. A laboratory-scale processing system has been developed using rapid prototype mold to fabricate rectangular gradient structures.

In summary, this research advances the understanding of design and fabrication process of graded metal-ceramic composites. The contributions made by this researcher will open new pathways to the ultimate goal of the commercial processing of products using graded metal-ceramic composites.

3.6 Material Selections

The fabrication of functionally graded materials requires careful matching of required constituents i.e. ceramic and metal powders. The firing treatment applied to compacting specimen must be kept 0.6 times the absolute melting points of the materials. As such, melting temperature acts as the upper limiting value in the solid-state sintering process. The material with the lower melting temperature limits the achievable maximum sintering temperature for the entire graded material structures. Sintering temperature relative to the melting point of the material is a critical factor to the amount of sinter bonding that occurs in the required structure. As a result, the material with the lower melting temperature does not reach the same extent of sinter bonding as the material with the higher melting temperature [8]. Due to the relatively low melting point of most metals, the choice of appropriate ceramics and metals are limited. Properties of Nickel and Alumina are shown in Table 1.

Table 1: Standard Properties of Alumina and Nickel [16].

Property	**Alumina**	**Nickel**
Composition	Al_2O_3	Ni
Crystal Type	Hexagonal	FCC
Density (g/cm^3)	3.9	8.9
Melting Temperature (˚C)	2050	1450
Elastic Modulus (GPa)	400	220
Poisson’s Ratio	.25	.30
Yield Strength (MPa)	300	130

Hardness (HV)	1500	40

3.7 Particle Size

In order to tailor composite microstructure, discrete layer reinforcement is needed. By controlling the particle sizes and size ratio between the matrix and inclusion phases, it is possible to get adequate sinter bonding in the ceramic-rich compositions and acceptable mechanical properties in the metal-rich compositions [13]. For notation purposes, the content of a compositional element will be denoted by volume percent of Nickel. Composite elements above 50 vol. % Nickel is metal matrix composites (MMC) with large Alumina reinforcing particles and elements below 50 vol. % Nickel is ceramic matrix composites (CMC) with large Nickel reinforcing particles. The particle sizes of all materials used in this research are presented in Table 2.

Table 2: material powders used for this research.

Material	**Type**	**Average Particle Size (μm)**
Nickel	123 HDNP	4 13
Alumina	RC-HP Gilox-63	0.5 15
TiO_2	Anatase Nanopowder	0.02

3.8 Nanopowder TiO_2

Anatase nanopowder TiO_2 was chosen to add as a sintering additive in part due to its easy availability and relatively cheaper price. In addition, previous work has indicated small quantities of TiO_2 can improve grain growth at lower temperatures in pure Alumina and reduce the sintering temperature [19, 20]. For this research, 3 wt. % TiO_2 was mixed with the pure Alumina for use in structurally graded compositions. TiO_2 percentage is constant in the Alumina at 3 wt. %, but the total weight percent of each composite composition falls with the rise of Nickel content and decreasing Alumina content. Consequently, no TiO_2 powder is added to the 100 vol. % Nickel powder (pure nickel powder only i.e. no alumina powder). The blending of powders was obtained by a conventional mixing process. Basic properties of TiO_2 are given in Table 3.

Table 3: Standard properties of TiO_2 [16].

Property	Anatase TiO_2
Density (g/cm^3)	4
Crystal Type	Tetragonal
Melting Temperature (˚C)	1850
Elastic Modulus (GPa)	280
Poisson’s Ratio	.27
Yield Strength (MPa)	92
Hardness (HV)	900

3.9 Binder

Binder is typically added to powders in very small quantities to the formation of unconsolidated green compacts. For this current research, a low-density organic binder Q-PAC

40 (polypropylene carbonate, $CH_3CH_2CH_2{\cdot}CO_3$, 1.3 g/cm^3) was used along with certain powder compositions. Previous work has examined that this particular binder completely burns out during the firing cycle, leaving little or no residual carbon [18]. Additionally, this binder was used to adapt compaction behavior and sintering performance in a thermal behavior. The binder burns out at low temperature and voids are left behind for the powder to consolidate, and the additional porosity accelerates densification process. When combined with the selection of particle size distribution, binder can be used to control three critical parameters: (1) sintering start temperature, (2) sintering rate, and (3) total shrinkage [18].

To apply the binder to the powders, Q-PAC 40 was added in acetone in a controlled weight percentage of the powder compositions in required quantities. These quantities are based on previous work to match the relative green density for the powder compositions [1, 18]. Binder quantities are given in Table 4 for different volume percentage of nickel.

Table 4: Binder quantities [18].

10	4
20	3.5
30	4
40	3.5
60	1
70	0
80	0
100	0

CHAPTER 4

Methodology for fabrication of experimental setup

To design and to fabricate a laboratory scaled experimental setup, the following techniques are followed by the analysis of the characterization of different material properties. In the previous chapter material size and different conditions required to design a Functionally Graded Material specimen are described, and structural gradations are studied. Here detailed fabrication techniques are presented to design the experimental setup by analysis of mechanical properties.

4.1 Quick Return Motion Mechanism (QRMM)

A four bar chain with a prismatic bar as a limiting case of a revolute pair and superimposed upon this is a kinematic inversion of the slider crank chain. Crank and slotted lever mechanism is the underlying mechanism used to spread metal-ceramic powders on the design mould by a quick return motion mechanism. This device is used in slotting machines, shaping machines and in case of internal combustion engines. In this device, the link 3 is fixed which forms the turning pair as shown in figure 4.1 and this link corresponds to connecting rod of a reciprocating engine. The driving crank (driven by a motor) CB revolves about fixed center C with uniform angular speed. A sliding plate is connected to the crank pin at B slides along the slotted bar AP and thus causes AP to oscillate about the pivot point A. A short link PR that transmits the motion from AP to the ram carries the tool and reciprocates along the line of stroke R1R2 that is perpendicular to AC to which cutting tool is attached. In the extreme position, AP1 and AP2 are tangential to the circle. The forward stroke (cutting stroke) occurs when the crank rotates from the position CB1 to CB2 in clockwise direction. The return stroke is produced when crank rotates from the position CB2 to CB1 in clockwise direction.

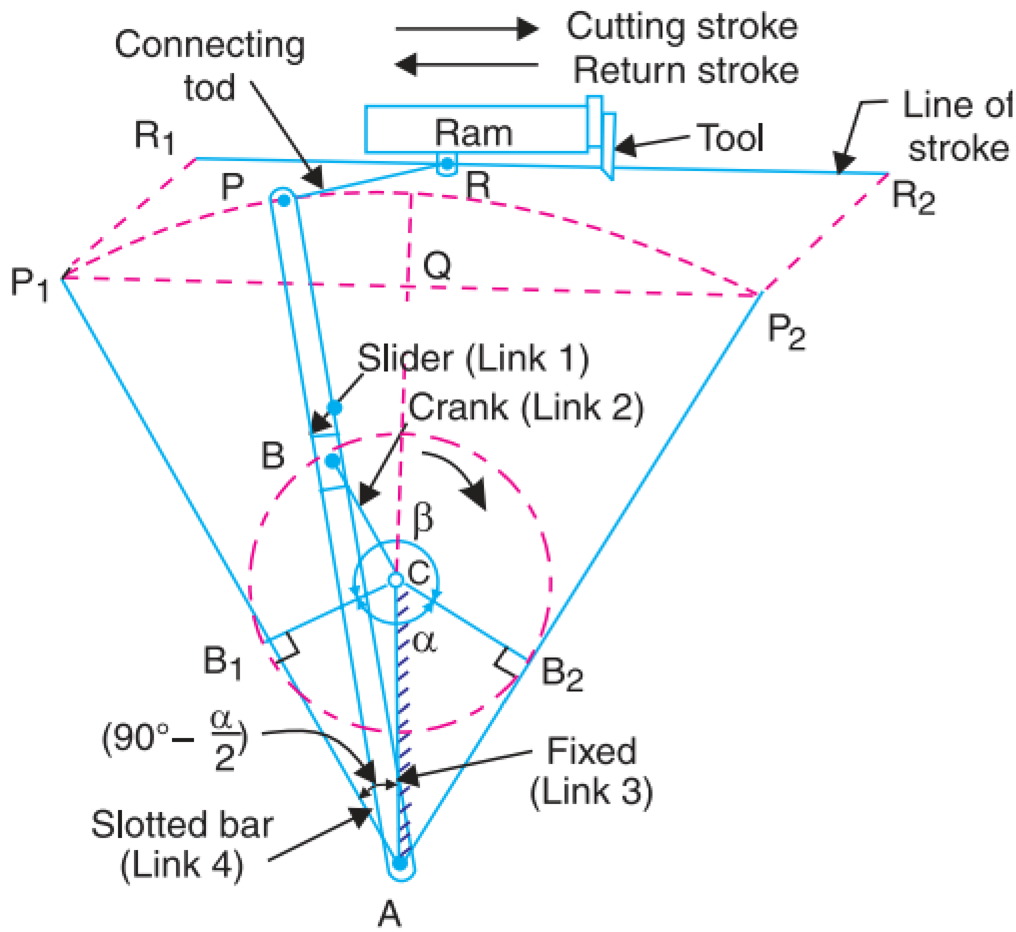


Figure 4.1: Crank and slotted lever quick return motion mechanism.

Data needed for modeling of crank slotted lever mechanism is presented in Table 1. The design has been prepared using the software CATIA V5R21. Dimensions of the mechanism given in the table are very useful for calculation of time ratio and length of the stroke. The modeling of different components of QRMM is explained here using CATIA software.

A slotted lever is connected to the crankshaft that provides the forward and backward motion of the tool post. The drawing is done as per dimensions by using CATIA V5R21 software that is shown in figure 4.2. The Crankshaft is the most sensitive link to that closer tolerance is provided to get the desired output and this link is connected to motor provided rotation of both crankshaft and slotted lever.

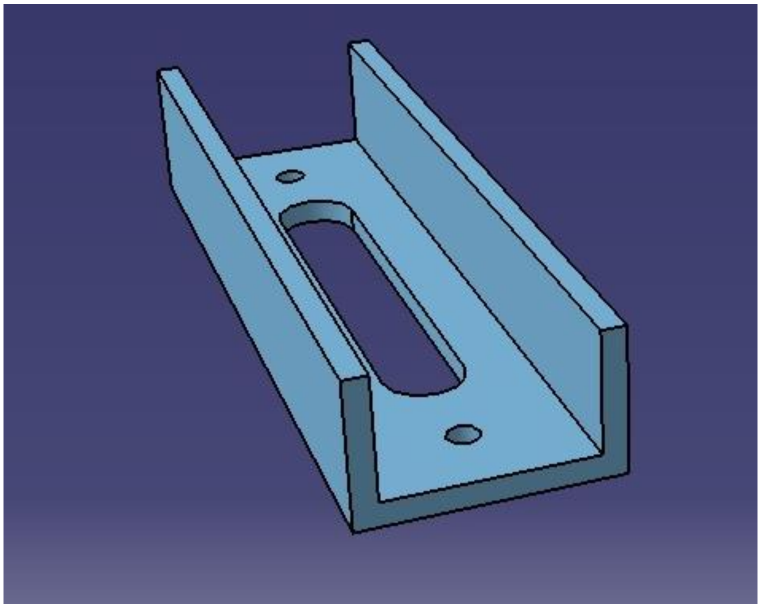


Figure 4.2: Slotted bar

The drawing is drawn as per the dimensions by using CATIA V5R21 software that is shown in figure 4.3.

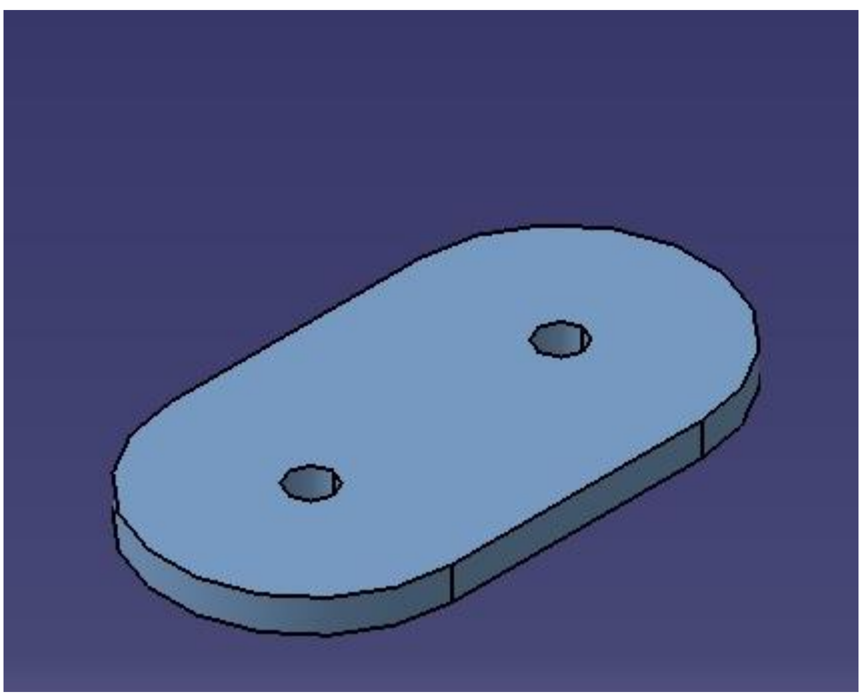


Figure 4.3: Crankshaft

The fixed link is rigidly fixed to the wooden stand of the FGMS setup and connected to the crankshaft and is designed by CATIA V5 R21 which is illustrated in figure 4.4. Connecting rod as shown in figure 4.5 is attached slotted bar that causes reciprocating motion to slider 2 (sliding area) as illustrated in figure 4.6. Slider 1 shown in figure 4.7 is connected to crankshaft and slides on the slotted lever bar. Rocker, as shown in figure 4.8 reciprocates as crankshaft rotates. A quick return motion mechanism is used to control discrete powder flow layering by using flow control valves. The Nozzle, as shown in figure 4.9 is used to flow of mixed powder with exact proportion required for layering to form functionally graded plate specimen. The powder falls from the nozzle end on the designed mould layer by layer to form FGMs plate as shown in figure 4.10.

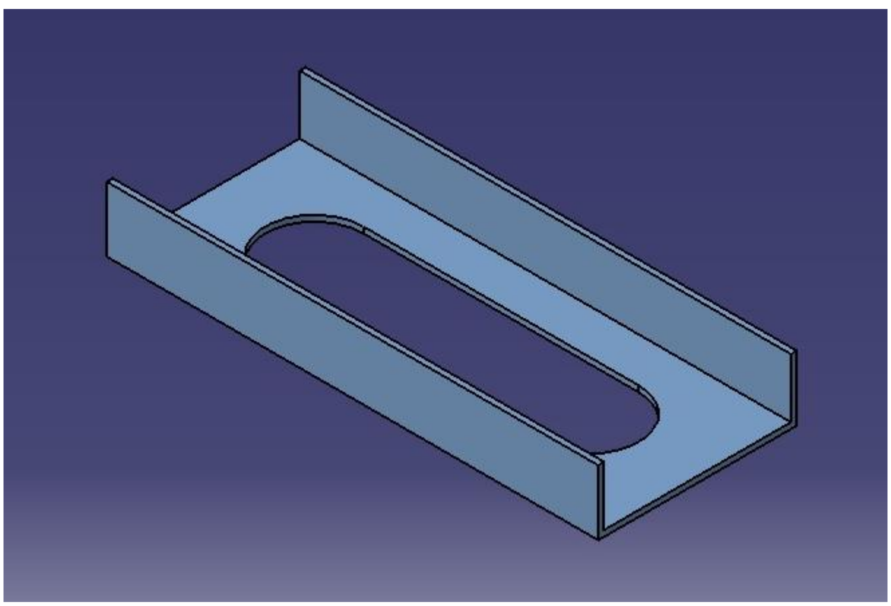


Figure 4.4: Fixed link

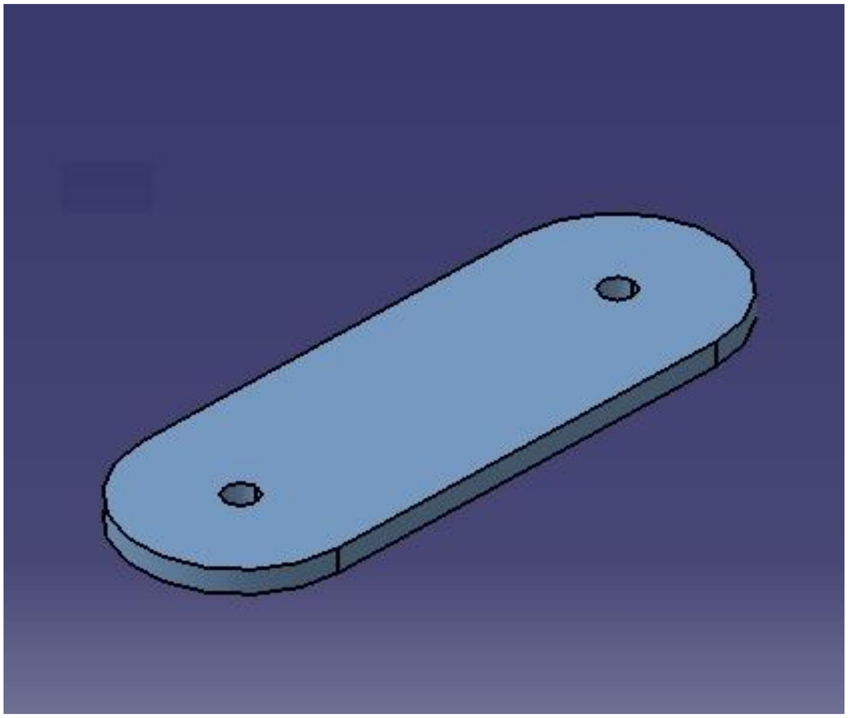


Figure 4.5: Connecting rod

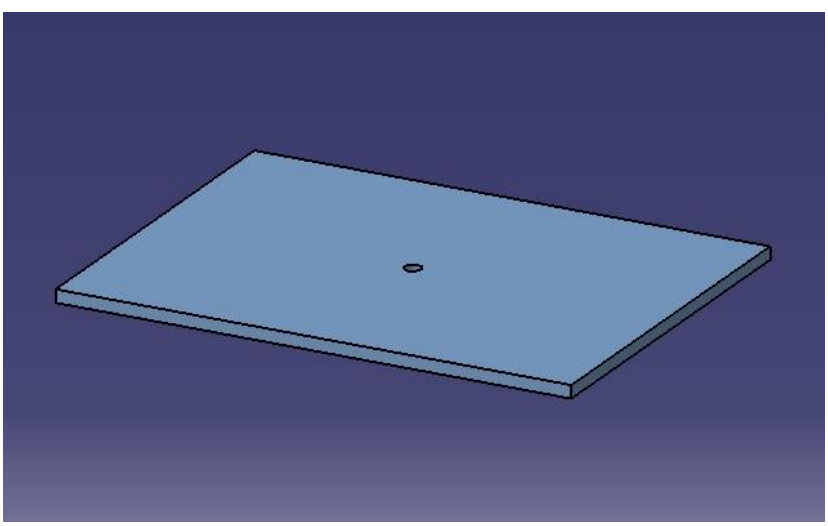


Figure 4.6: Slider 2

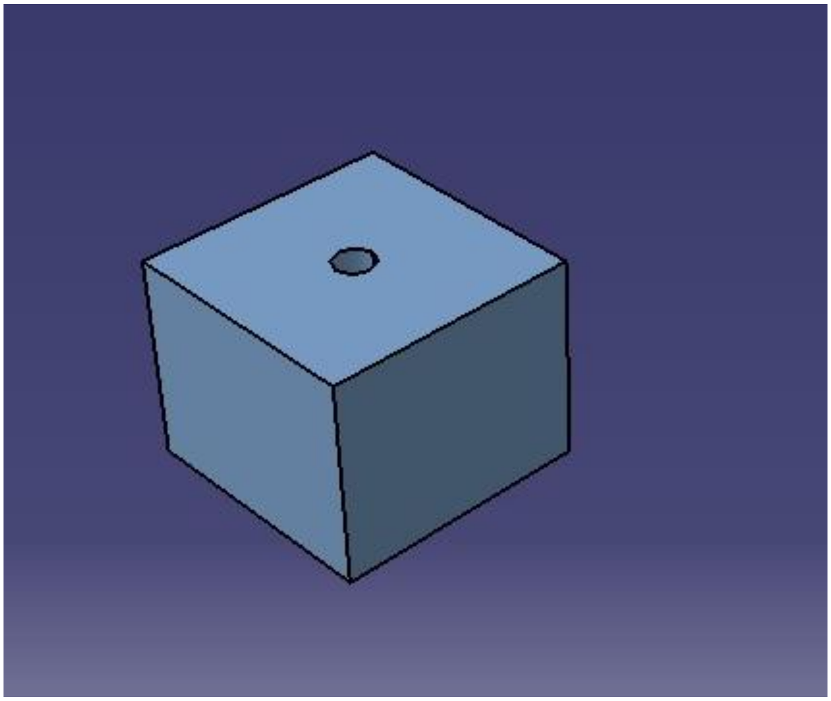


Figure 4.7: Slider 1

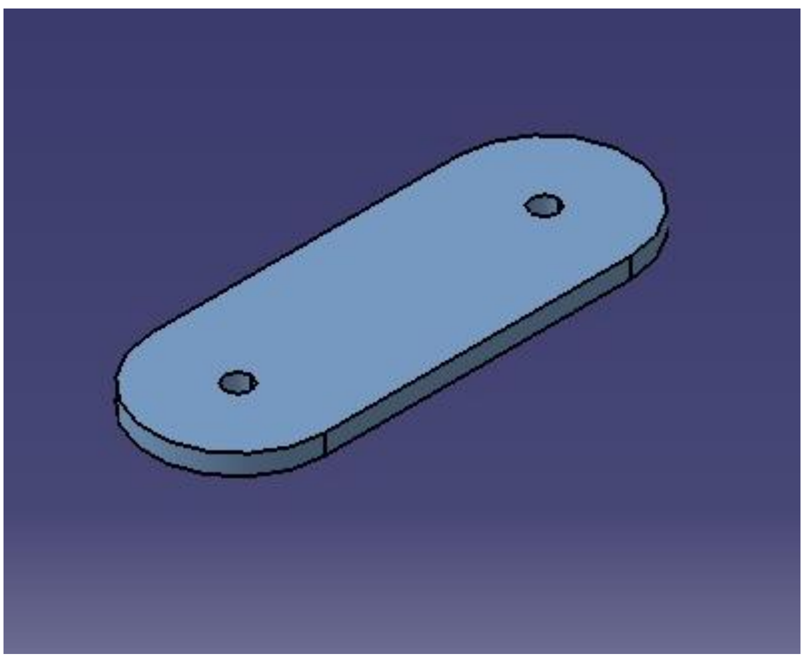


Figure 4.8: Rocker

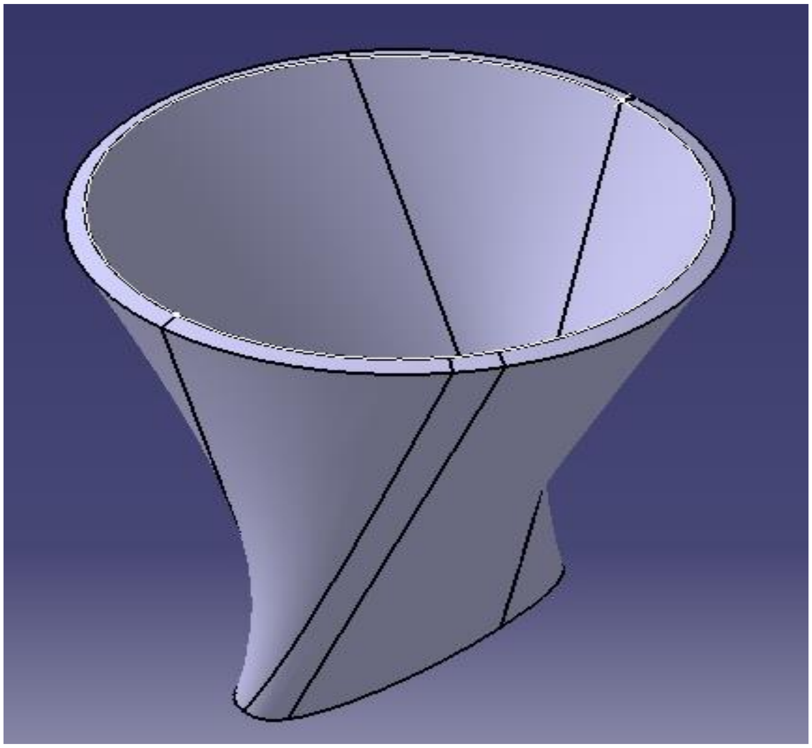


Figure 4.9: Nozzle

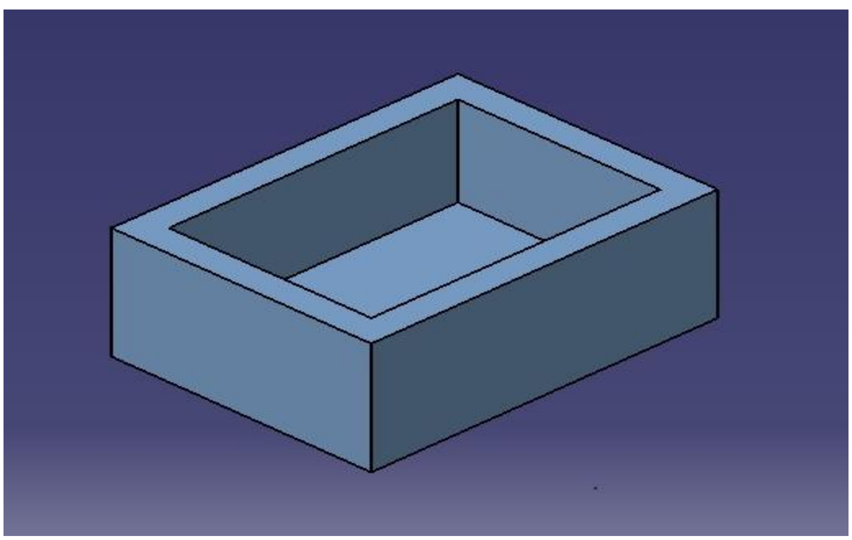


Figure 4.10: Moulding box

Different link dimensions that are required for crank slotted lever (quick return motion mechanism) design are given in table 5.

Table 5: Dimensions of links of crank slotted lever mechanism

SL No.	Links	Dimensions(in mm)
1	Crank	23*26*3
2	Fixed link	46*26*3
3	Slotted lever	80*45*3
4	Slider 1	25*25*20
5	Slider 2	120*90*3
6	Connecting rod	50*26*3
7	Moulding box	80*50*5

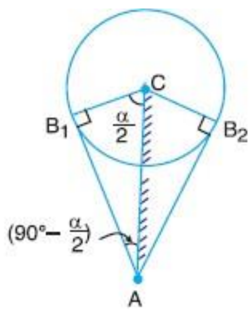
4.2Fabrication procedure of experimental setup

All parts of quick return motion mechanisms (fixed link, crankshaft, slotted bar, connecting rod, slider 1, slider 2, rocker) are fabricated using rapid prototyping technology with proper dimension by ABS materials. Nozzle and mould are also fabricated using rapid prototyping technology. Rapid prototyping technology is an additive manufacturing process with good accuracy where complex parts like nozzle can be fabricated without any loss of materials. This technology is chosen to avoid complexity of manufacturing and to reduce weight. Quick return motion mechanism parts are assembled using 4mm nut screw and bolt then a 60 rpm dc geared motor is attached to the crankshaft to provide rotation to the mechanism. The shaft of geared motor is entered in the crankshaft by drilling and then motor is fixed by use of a metallic stand and pins. A wooden stand is attached to fixed link of mechanism and mechanism is placed on wooden base support. The nozzle is fixed on the above of the QRMM by a wooden support. Both Pipes are sealed to two chutes for two different powder flow alumina and nickel respectively. To control powder flow mechanically, two flow control valves are attached to both pipes. After mixing in exact ratios flow through third pipe occurs which also controlled by another flow control valve. The powder will fall during forward stroke of QRMM by controlling the flow control valve and during return stroke it will remain closed. To control flow of powder 90-degree rotation valve is used. On this valve for each degree of rotation percentage of powder flow is marked by sketch. Finally, laboratory scaled FGMs setup to fabricate a particular specimen is fabricated.

Layer by layer powder will fall into the mould after that it will be compacted using hand driven rectangular metallic plate for compaction and then sintered to get final specimen.

4.3 Determination of Time ratio and stroke length

Let $\angle CAB_1$ = Inclination of the slotted bar with the vertical.



We know that

$\sin \angle CAB = \sin (90^0 - \frac{\alpha}{2})$

$$\frac{B1C}{AC} = \frac{23}{46} = 0.5$$

$\angle CAB = (90° - \frac{\alpha}{2}) = 30^0$

Again,

$\angle CAB = (90° - \frac{\alpha}{2})$

Hence, $\alpha = 120^o$

Time ratio =

$$\frac{Time\ of\ Cutting\ Stroke}{Time\ of\ Return\ Stroke} = \frac{360-\alpha}{\alpha} = \frac{360-120}{120} = 2$$

Length of stroke = $R_1R_2 = P_1P_2 = 2\ P_1Q$

$= 2\ AP_1 \sin (90° - \frac{\alpha}{2})$

$= 2 * 80 * 0.5 = 80$ mm

CHAPTER 5

Results and discussions

In this thesis paper it is discussed that nickel and alumina along with titanium oxide as sintering aid are primary components for design of FGMs specimen. To design a rectangular plate type specimen a laboratory scale based Functionally Graded set up was fabricated. For this fabrication different parts of quick return motion mechanisms are fabricated. Crank, fixed link, rocker, connecting rod, slider 1, slider 2, nuts, bolts, dc geared motor, nozzle, mould box, mechanically operating valves, pipes and funnel are assembled and frame is designed using wooden piece. The complete set up for laboratory scale functionally graded material is as shown below.

Figure 5.1. Functionally Graded Material Setup

CHAPTER 6

Conclusions

The novelty reported in this thesis focused on the development of a set up for design of functionally graded materials by using techniques such as powder processing techniques, rapid prototyping and to facilitate the pressureless sintering of metal-ceramic gradient structures. A new nanoparticle sintering aid was used to Nickel-Alumina composites to control mechanical properties during fabrication in a pressureless sintering process. Functionally graded structures were characterized by changes in microstructure, porosity, and micro-hardness. Firstly, design is completed of different links (crank, slotted lever, fixed link, slider 1, slider 2, connecting rod), nozzle and Moulding box using CATIA. Then all designed parts are fabricated using rapid prototyping (RP) technology. All link lengths and timing ratios are calculated for the required specimen. Simulation is completed first using CATIA software then quick return mechanism is operated by geared motor successfully. Pipes, nozzle, nuts, bolts, quick return motion mechanism are assembled perfectly and a frame is fabricated. Finally, a laboratory- scale based set up is fabricated and machine can be automated using Adriano. By required powder composition FGMs of metal-ceramic with rectangular specimen can be fabricated.

CHAPTER 7

Research contributions and Future Work

This research advances the understanding of the processing techniques of pressureless sintered graded metal-ceramic composites. The main scientific and technical contributions of this research are as follows:

[1] For the first time, a nanoparticle sintering aid has been utilised to a wide range of pressureless sintered metal-ceramic homogeneous composites and metal ceramic gradient structures. The resulting change in micro hardness, porosity and microstructures are characterized.

[2] For the first time, concrete molding technology has been adapted for the fabrication of graded metal-ceramic composites in geometrically complex structures. The developed experimental set up for gradation was fabricated and to control flow of powders mechanical valve was fitted and manually controlled with proper regulation as marked on the valve.

[3] For the first time, the bulk processing of graded metal-ceramic composites in geometrically complex structures has been investigated. Using powder metallurgy and rapid prototyping techniques, a laboratory-scale processing assembly has been developed to design a graded rectangular plate specimens.

The processing of pressureless sintered metal-ceramic composites continues to present unresolved fabrication issues. The modified thermal-behavior matching process developed by this research, using particle size distributions, binder additives, and nanoparticle sintering aids, should be further refined to better results. Indeed, the poor sintering characteristics of certain compositions due to particle agglomerates, particularly in the 60 vol. % Nickel layer, must be improved. An attempt must also be made to better match the sintering of pure Nickel, which sinters faster and more completely than any other composition

CHAPTER 8

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